

**WE CLAIM:**

1. A process for isolating and purifying phytosterols and phytostanols from tall oil pitch  
5 which comprises:

a) feeding the pitch into a first distillation column;

b) distilling the pitch to remove excess rosin acids and fatty acids to form a distilled  
pitch;

10 c) saponifying the distilled pitch with an aqueous solution of one or more alkali metal  
bases to form a saponified pitch;

d) neutralizing the saponified pitch with an amount of acid sufficient to achieve an  
ending pH of between 5.8 and 6.3 thereby forming a neutralized pitch;

15 e) allowing the neutralized pitch to phase separate for a period of at least 12 hours or  
until the water content of the pitch, on phase separation, is less than 15%, thereby  
forming a settled pitch and a water phase;

f) removing substantially all of the remaining water from the settled pitch to form a  
modified pitch;

20 g) distilling the modified pitch in a second distillation column to remove lights ends from  
the modified pitch and to produce a bottom fraction comprising free phytosterols and/or  
phytostanols;

h) distilling only the bottom fraction in a third distillation column to produce a light  
phase distillate comprising free phytosterols and/or phytostanols;

i) dissolving only the light phase distillate in a solvent comprising at least one alcohol to  
produce a solution of phytosterols and/or phytostanols;

25 j) cooling the solution to form a slurry with phytosterols and/or phytostanols crystallized  
therein; and

k) washing, filtering and drying the slurry to isolate the crystallized phytosterols and/or  
phytostanols from the filtrate.

2. The process of claim 1 wherein, in step b), the pitch is distilled to achieve an acid value of less than 40.

5 3. The process of claim 1 wherein, in step b), the pitch is distilled to achieve an acid value of less than 30. <sup>7</sup>

4. The process of claim 1 wherein the distillation columns in steps b), g) and h) are selected from the group consisting of short path distillation columns, wiped film  
10 evaporation columns, thin film evaporation columns and molecular distillation columns.

5. The process of claim ~~4~~ wherein the distillation column in steps b), g) and h) is a wiped film evaporation column.

15 6. The process of claim 1 wherein there is provided an additional, concurrent feed into the first distillation column, said feed being the filtrate from step k), characterized in that the filtrate is pre-treated to strip it of solvents and to convert substantially all the free sterols therein to steryl esters.

20 7. The process of claim ~~1~~ wherein, in step d), the saponified pitch is neutralized at a temperature exceeding 100°C for a period of from 1 to 10 hours.

8. The process of claim 1 wherein steps c) and d) occur in the same reaction vessel.

25 9. The process of claim 1 wherein step d) is carried out under vigorous agitation.

10. The process of claim 1 wherein in step d) the acid and pitch are mixed.

11. The process of claim 1 wherein step e) is carried out without agitation.

12. The process of claim 1 wherein there is provided an additional step, after step e) which comprises subjecting the water phase to a second phase separation.

13. The process of claim 1 characterized in that the step of removing substantially all of the remaining water from the settled pitch in step f) to form a modified pitch comprises the use of a water strip wherein the pressure is no greater than atmospheric and the temperature is below 105° C.

14. The process of claim 13 wherein:

- a) the temperature is cooled to 80° C or less after the water strip; and
- b) the temperature is further cooled to 60° C or less if the modified pitch is to be stored prior to initiation of step g).

15. The process of claim 1 wherein the alkali metal base is selected from the group consisting of sodium hydroxide, potassium hydroxide or combinations of both.

16. The process of claim 1 wherein the mineral acid is selected from the group consisting of sulfuric acid, hydrochloric acid, phosphoric acid or any combination thereof.

17. The process of claim 1 wherein the solvent in step i) comprises a low molecular weight monohydric alcohol or an acetate ester thereof.

18. The process of claim 17 wherein the alcohol is selected from the group consisting of methanol, ethanol and isopropanol and acetate esters thereof.

19. The process of claim 1 wherein the solvent in step i) is selected from the group consisting of ketones and C1 to C8 hydrocarbons, or mixtures thereof.

20. A composition prepared by the process of claim 1.

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